

**ATTORNEY DOCKET NO. FA/264**

**UNITED STATES PATENT APPLICATION**

**OF**

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**AND**

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**FOR**

**NOVEL POLYMER FILMS AND TEXTILE LAMINATES  
CONTAINING SUCH POLYMER FILMS**

**TITLE OF THE INVENTION**

Novel Polymer Films and Textile Laminates Containing Such Polymer Films

**FIELD OF THE INVENTION**

5           This invention relates to polymer films and laminates comprising such films and at least one textile material that provide, among other things, improved aesthetics and methods for making such films and laminates.

**BACKGROUND OF THE INVENTION**

10           Porous, expanded polytetrafluoroethylene (hereinafter ePTFE), made by expansion by stretching at a temperature below the crystalline melt temperature of PTFE, has been known for some time. These porous, fibrillated materials and their manufacture were originally described by Gore in U.S. Pat. Nos. 3,953,566 and 4,187,390. They possess the known attributes of PTFE while adding  
15 additional benefits resulting from their porous microstructure. They are typically hydrophobic, inert, water resistant, strong, and can be made to be thin and flexible. Applications for these materials include, for example, wire insulation, gaskets, and waterproof and breathable rainwear.

          The mechanical properties of these materials have been the focus of  
20 much research and many inventions. The use of elongation at high rates and elevated temperatures to increase the tensile strength of PTFE was first taught by Gore in U.S. Pat. No. 3,953,566.

          The creep resistance of ePTFE has been shown to increase by densification at high temperatures under vacuum by Knox, et al. in U.S. Patent  
25 No. 5,374,473. Densification of at least two layers of ePTFE was performed under vacuum at temperatures from 330°C to 390°C and under pressures from 150 psi to 350 psi.

          Likewise, creep resistant articles of ePTFE such as gaskets, O-rings, and valve seats have been produced under similar extreme process conditions. Fuhr, et.al., in U.S. Patent No. 5,792,525, teach that densification of ePTFE in an  
30 autoclave at temperatures over 300°C for an hour or more will significantly reduce the creep of the resultant ePTFE articles. In addition, Fuhr et al. teaches that partial densification results in an ePTFE structure that has a dense surface skin and a non-densified core.

35           The cut resistance of ePTFE articles has also been improved by densification at high temperatures. U.S. Patent No. 4,732,629 teaches that densification and heating ePTFE wire-wrapping tapes to over 345°C for a period

of time increases the cut resistance of the resultant cable shield by more than 50%.

Complimentary layers have been combined with ePTFE films to enhance specific performance attributes. The addition of a layer of hydrophilic polymer to the ePTFE film was first taught by Gore in U.S. Patent No. 4,194,041. This patent showed that the contamination resistance of waterproof, breathable articles, such as garments, could be enhanced by the addition of a layer of breathable polymer to an ePTFE film. It is also known to sandwich such a complimentary layer between two or more layers of ePTFE film, or to provide a complimentary layer to both sides of the ePTFE film.

It is further known to form ePTFE containing laminates, which include at least one layer of ePTFE film adhered directly to at least one textile material. Moreover, the ePTFE film can contain the above mentioned layer of hydrophilic polymer, if desired. In such an embodiment, the hydrophilic polymer layer can serve to adhere together the ePTFE film and the textile material, or a suitable adhesive material can be supplied to adhere the hydrophilic polymer and ePTFE combination to the textile material. Moreover, in the case where a hydrophilic polymer layer is not provided, an adhesive material can be supplied to adhere the ePTFE film to the textile material. Any suitable adhesive can be used. The adhesive can be supplied in a discontinuous pattern (e.g., as a discreet dot pattern, as lines of adhesive, etc.), as a substantially continuous layer (e.g., substantially covering the surface of the ePTFE), in which case the adhesive should be capable of allowing water vapor to pass through (i.e. be breathable, such as the hydrophilic polymers mentioned above, and particularly hydrophilic layers of polyurethane), or the adhesive can be supplied as a continuous pattern, such as the grid pattern disclosed in US Patent No. 5,660,918, to Dutta.

All of the above teachings have lead to the development of ePTFE films and laminates which are ideally suited for protective clothing articles used for wear in wet conditions (such as rain, snow, etc.); in outdoor activities (such as skiing, biking, hiking, etc.); in handling hazardous chemicals, in preventing contamination, in avoiding infection, articles should in each instance protect the wearer by preventing leakage of water or other fluids and microorganisms into the article while keeping the wearer comfortable by allowing perspiration to evaporate from the wearer to the outside of the article. In addition, such articles are intended to be reusable. That is, it should maintain the functional attributes of protection and comfort during ordinary use including automatic machine washing.

The microporous structure of ePTFE make these films inherently white in color. For certain garment and other applications, this white color can be aesthetically displeasing and thus highly undesirable. U.S. Patent No. 3,953,566 teaches that ePTFE film can be made transparent if heat treated at 350°C for 8 minutes and then compressed under 1500 psi for several minutes. This means of creating a non-white ePTFE microstructure fails to meet the needs for breathable constructions because the process used to create the transparency simultaneously results in a fully densified, non-porous film.

Alternatively, colored fillers have been used to uniformly change the inherent white color of ePTFE. U.S. Patent No. 4,985,296, teaches the use of fillers such as carbon black to create homogenous ePTFE films of colors other than white.

Unfortunately, the fact that all ePTFE film containing laminates to date have a uniform, single color appearance forces the garment manufacturers to hide the white ePTFE film either by including a third textile layer on the inside surface of the garment, or by using a three-layer laminate wherein the ePTFE film is sandwiched between two textile layers. Thus, a need exists for an ePTFE containing laminate that is not a single homogenous color but rather provides an aesthetically pleasing, patterned appearance.

## **SUMMARY OF THE INVENTION**

Polymer film having at least two regions of differing translucency is provided.

Laminate comprising at least one layer of textile material having a first side and a second side and polymer film, having a first side and a second side, adhered to at least one of the first and second sides of the textile material, wherein the polymer film has at least two regions of differing translucency is also provided.

The at least two regions of differing translucency can be obtained by a process where regions of the polymer film are selectively compressed, and other regions are not compressed. Such a process can comprise compressing a polymer film or a polymer film/textile laminate between two hardened surfaces, at least one of which may contain a pattern that is to be imparted into the polymer film. The two surfaces are brought together with sufficient pressure to effectively compress only the portions of the polymer film where the pattern is raised while concurrently retaining non-compressed portions where the pattern is not raised. Heat can optionally be applied to effect the contrast between the compressed and non-compressed portions of the desired pattern. Patterns can be

created by using at least one hardened surface comprising multiple levels to which the raised surface is raised. Each different raised level can create a correspondingly different amount of compression (and, thus, degree of translucency) of the polymer film. As the polymer film is compressed, it becomes more translucent, as compared to the non-compressed (or less compressed) portion of the film. The degree of translucency increases with increasing level of compression. Therefore, selective compression accomplished through the use of patterned raised surfaces can create aesthetically enhanced polymer films and polymer film/textile laminates. Due to the optical change (i.e., increased translucency) of the polymer film upon compression, the image of the patterned surface can be transferred to the polymer film or polymer film/textile laminate.

### **BRIEF DESCRIPTION OF THE FIGURES**

Figure 1 is a schematic drawing of a process according to the invention;  
Figure 2 is a schematic drawing of a process according to the invention;  
Figure 3 is a schematic drawing of a process according to the invention;  
Figure 4 is an optical micrograph of a laminate according to the invention;

Figure 5 is an optical micrograph of a laminate according to the invention;

Figure 6 is an optical micrograph of a laminate according to the invention;

Figure 7 is a combination of an optical micrograph and a corresponding grey-scale spectra showing the range of shades of grey in a laminate according to the invention;

Figure 8 is an optical micrograph of a laminate according to the invention;

Figure 9 is a combination of an optical micrograph and a corresponding grey-scale spectra showing the range of shades of grey in a laminate according to the invention;

Figure 10 is an SEM of a laminate according to the invention;

Figure 11 is a three-dimensional surface map depicting the various depths of a laminate according to the invention;

Figure 12 is a Zygo profilometer scan of the surface of a laminate according to the invention;

Figure 13 is an optical micrograph of a section of a prior art laminate that has been subjected to abrasion testing; and

Figure 14 is an optical micrograph of a section of a laminate according to the invention that has been subjected to abrasion testing.

### **DETAILED DESCRIPTION OF THE INVENTION**

5 Polymer film having at least two regions of differing translucency is provided.

Laminate comprising at least one layer of textile material having a first side and a second side and polymer film, having a first side and a second side, adhered to at least one of the first and second sides of the textile material, 10 wherein the polymer film has at least two regions of differing translucency is also provided. In an aspect of the invention the polymer film comprises a multitude of regions differing in translucency. In a further aspect of the invention the regions of differing translucency comprise a substantially repeating pattern. In a further aspect of the invention, at least about 10% of the 15 surface area of the polymer film is more translucent than the remaining surface area of the polymer film. In a still further aspect of the invention, at least about 25% of the surface area of the polymer film is more translucent than the remaining surface area of the polymer film. In yet a further aspect of the invention, at least about 50% of the surface area of the polymer film is more 20 translucent than the remaining surface area of the polymer film.

As used herein "polymer film" includes any polymer structure whose optical properties (i.e. translucency) change upon compression. In an aspect of the invention "polymer film" can be a polymer film having at least some interconnected porosity. Examples of suitable polymer films include porous 25 polytetrafluoroethylene films (and particularly porous, expanded polytetrafluoroethylene films ("ePTFE")), porous polyurethane films, porous polyolefin films, porous polyester films, and multi-colored polyurethane films. Although polymer films can be any polymer structure, as discussed above, for simplicity the remainder of the disclosure will exemplify an embodiment 30 wherein the polymer film comprises porous, expanded polytetrafluoroethylene film.

Suitable textile materials may be woven or non-woven, employing synthetic fibers, natural fibers, or blends of synthetic and natural fibers. Textiles may also be knits, interlocks and brushed knits.

35 Laminate can be formed by the selective compression of an ePTFE film or of an ePTFE film/textile laminate which will result in, among other things, improved aesthetics while other attractive properties (such as breathability) are not significantly affected. A selectively compressed ePTFE film and/or ePTFE

film/textile laminate can be produced by a number of suitable methods. ePTFE film/textile laminates provide a useful material for many applications where water vapor permeability (i.e., breathability) is required while providing some degree of water resistance or even water proofness.

5           Laminates comprising ePTFE film and textile can be produced by any suitable method. Such methods are known in the art and include those as described in, for example, U.S. Patent No. 5,289,644 to Driskill et al. For example such laminates can be produced by printing an adhesive onto one layer in a discontinuous pattern, in an intersecting grid pattern, in the form of  
10 continuous lines of adhesive, as a thin continuous layer, etc., and then introducing the second layer in a way that the adhesive effectively joins and adheres together the two adjacent surfaces of the ePTFE film and the textile material. A 3 layer laminate can similarly be produced by printing adhesive onto both sides of an ePTFE film and then introducing both a first and second  
15 textile to opposing surface of the ePTFE film onto which the adhesive has been printed. Alternately, a 2 layer laminate can be produced first and then an adhesive printed or otherwise provided onto the ePTFE side of the 2 layer laminate prior to the introduction of a second textile layer onto said second ePTFE surface.

20           The at least two regions of differing translucency can be obtained by a process where regions of the polymer film are selectively compressed, and other regions are not compressed. Such a process can comprise compressing a polymer film or a polymer film/textile laminate between two hardened surfaces, at least one of which may contain a pattern that is to be imparted into the  
25 polymer film. The two surfaces are brought together with sufficient pressure to effectively compress only the portions of the polymer film where the pattern is raised while concurrently retaining non-compressed portions where the pattern is not raised. Heat can optionally be applied to effect the contrast between the compressed and non-compressed portions of the desired pattern. Patterns can be  
30 created by using at least one hardened surface comprising multiple levels to which the raised surface is raised. Each different raised level can create a correspondingly different amount of compression of the polymer film. As the polymer film is compressed, it becomes more translucent, as compared to the non-compressed (or less compressed) portion of the film. The degree of  
35 translucency increases with increasing level of compression. Therefore, selective compression accomplished through the use of patterned raised surfaces can create aesthetically enhanced polymer films and polymer film/textile laminates. Due to the optical change (i.e., increased translucency) of the

polymer film upon compression, the image of the patterned surface can be transferred to the polymer film or polymer film/textile laminate.

Turning to the Figures, Figure 1 is a schematic of a process by which selective compression of an ePTFE film/textile laminate can be accomplished.

5 As shown in Figure 1, an ePTFE film 2 has been laminated to textile material 3. The laminate can be calendered between two smooth hard rolls 1 and 4 under pressures, temperatures, and speeds so that the raised yarns of the textile are sufficient to locally compress the ePTFE microstructure; thereby creating a selectively compressed ePTFE film/textile laminate. In both woven and knitted  
10 textiles, the plies of the yarns can be alternated one over the other. One yarn can be raised and then descend under the other yarn as the textile is intertwined together which creates surface topography. As the ePTFE laminate is subjected to pressure (and heat when used), such as when compressed between the two smooth rolls, the raised cross-overs of the yarns create high pressure spots that  
15 result in selective compression of the ePTFE microstructure, which results in the compressed regions of the film being more translucent than the non-compressed regions of the film. Furthermore, the pattern of selective compression in this case will match that of the textile used to create the high pressure points.

A 3 roll lab calendering system, from B.F Perkins of Rochester, N.Y. is  
20 an example of a suitable calendering system which can be used for such a method. The machine has a 22" wide face and can be run at between about 10 feet per minute and about 15 feet per minute in most cases. However, the speed with which ePTFE film/textile laminate can be passed through the nip of the smooth rolls can be altered greatly, depending on desired results. For example,  
25 higher line speeds will reduce the residence time under the pressure of the nip and accordingly will, generally, tend to produce less selective compression. Moreover, the temperature of the rolls can be adjusted to affect the degree of selective compression. Particularly suitable roll temperatures may be between about 80°C and about 160°C. However, a selective compression image can be  
30 created at both lower and higher temperatures. The nip pressure between the two smooth rolls can be varied, with pressures between about 250 pounds per linear inch and about 6,000 pounds per linear inch being particularly suitable. At low pressures, the intensity of the selectively compressed image may be lower. As the nip pressure is increased, the resultant selectively compressed  
35 image may become sharper up to a point, beyond which, the image may become more diffuse.

A second method for the selective compression of ePTFE film or ePTFE film/textile laminates is through the use of a pattern roll. A schematic of this



process configuration is depicted in Figure 2. In such a method, a pattern of high and low areas is created on an otherwise smooth roll. Typically, a hardened metal roll is engraved with the desired pattern 1'. This patterned roll 1' can then be matched with smooth, hard roll 4. The smooth roll 4 is typically  
5 comprised of hard rubber, plastic, or a filled fiber such as a filled cotton. A filled cotton roll is comprised of cotton fibers with an optional binder which are compressed to form a hard, packed solid material. This material can then be machined smooth to produce a suitable surface for smooth calendering. This pair of rolls can then be mounted in a machine so that a nip is formed between  
10 the rolls as depicted by the gap between 1' and 4 in Figure 2. The desired ePTFE film (or ePTFE film 2/textile 3 laminate) can then be fed through the nip that is formed between the patterned roll 1' and the smooth roll 4. As the ePTFE film (or ePTFE film 2/textile 3 laminate) passes through the closed nip, the pattern can selectively compress the ePTFE microstructure where the pattern surfaces  
15 are raised. This results in the compressed regions being more translucent than the non-compressed regions.

Suitable process roll temperatures can be between about 300°F and about 400°F, although both higher and lower temperatures can also produce selectively compressed ePTFE microstructures. The nip pressure can be varied  
20 depending on the desired degree of compression. Nip pressures of between about 10 and about 2000 pounds per linear inch ("pli") are believed to be particularly acceptable. In an aspect of the invention nip pressures of between about 100 pli and about 1000 pli may be acceptable. In a further aspect of the invention nip pressures of between about 200 pli and about 800 pli may be  
25 acceptable. The gap between the two compression rolls should be set to impart the desired nip pressure. Likewise, the line speed controls the residence time in the nip. Thus, slower line speeds will enable the ePTFE article to be subjected to the nip environment for a longer time as compared to when higher line speeds are used. In an aspect of the invention a line speed of between 1 yard per minute  
30 ("ypm") and 50 ypm can be used. In a further aspect of the invention, a line speed of between 5 ypm and 30 ypm can be used. In a still further aspect of the invention a line speed of between 10 ypm and 20 ypm can be used.

A third process for the selective compression of ePTFE film or ePTFE film/textile laminate is through the use of two pattern rolls as depicted in Figure  
35 3. In this process, the high and low portions of each pattern roll 1' and 1'' are synchronized so that the two rolls effectively mate with each revolution. This male/female set creates an enhanced, selective compression pattern. The intensity of the pattern can be adjusted based on the tolerances between the two

mating parts. The extent of selective compression imparted to the ePTFE film or ePTFE film/textile laminate depends in part on the gap between the two mating rolls, the temperature and pressure of the rolls, as well as the speed with which the article passes through the nip. As shown in Figure 3, while this process can be used on ePTFE film alone or on a 2 layer ePTFE film 2/textile 3 laminate, the process can also be used on 3 layer laminate of textile 3 /ePTFE film 2/textile 5, as shown in the Figure.

A fourth process for the creation of selectively compressed ePTFE film or ePTFE film/textile laminates is through the selective compression of an ePTFE film followed by lamination of the selectively compressed ePTFE to at least one textile material. In this process, an ePTFE film can be compressed between either a patterned surface and a smooth surface or between two patterned surfaces, as described above. If two patterned surfaces are used, these two surfaces do not need to be the same. In practice, this compression can be produced by passing the ePTFE film between the nip which is formed between either one patterned roll and one smooth roll or comprised of two pattern rolls.

Other suitable processes for selectively compressing polymer film or polymer film/textile laminates will now be readily apparent to the skilled artisan. The resultant material is not only aesthetically pleasing, but has other surprising, improved properties, as compared to a non-compressed polymer film or polymer film/textile laminate. For example, selectively compressed ePTFE film/textile laminate will remain breathable and will also compact better than a similar, but non-compressed laminate. Furthermore, selectively compressed ePTFE film/textile laminate will result in an ePTFE film layer that resists scratching, as compared to a similar, but non-compressed laminate. Moreover, selectively compressed ePTFE film/textile laminate has better (i.e. softer) "hand" (as described herein), than a similar, but non-compressed laminate.

The selectively compressed polymer film/textile laminates will have many useful applications as the skilled artisan will now understand. Exemplary articles that can be produced using the laminates include, for example, garments such as shirts, pants, gloves, shoes, jackets, vests, hats, etc. Further articles include, for example, tents, sleeping bags, etc. Such articles can be produced so that the polymer film faces outward and is the outer surface of the article. Moreover, such articles can be produced so that the polymer film faces inward (such as the inside surface of a garment) and is the inner surface of the article, thus obviating the need for an inner textile layer. Moreover, garments can be engineered to be fully reversible, so that the polymer film can face either outward or inward.

## **DEFINITIONS**

“Breathable” refers to polymer film/textile laminates that have a Moisture Vapor Transmission Rate (MVTR) of at least about 1,000 (grams/(m<sup>2</sup>)(24 hours)).

5           “Selective compression” refers to any process by which a region of the polymer film is compressed relative to a second region within the same specimen, resulting in the compressed region being more translucent than the non-compressed region. Thus, a selectively compressed polymer film will have at least two regions of differing translucency.

10           “Laminate” refers to any layered composite that comprises at least one polymer layer and at least one textile layer, the layers of which are, typically, adhered together.

          By “Adhered” or “Adhered together” it is meant that the polymer material (e.g., ePTFE film) and textile material are joined together by suitable bonding media. The bonding media can be adhesive dots, adhesive applied as a continuous grid pattern, adhesive applied as continuous lines, a continuous, breathable adhesive layer, a fusion bonded interface, or any other material which provides for adhesion between the textile layer and the polymer layer. In an aspect of the invention at least one layer of material (typically hydrophilic polymer) can be provided between the polymer film and the textile material. For example, a thin layer of hydrophilic polyurethane can be provided to the surface of an ePTFE film before the film is adhered to a textile material. Suitable adhesive can then be applied to the breathable polyurethane layer and then joined to the textile material. According to the invention the ePTFE film and the textile are considered adhered together even though at least one layer of material (such as hydrophilic polymer) is provided between them, in addition to the adhesive. In a further aspect of the invention the at least one layer of material can act as both a hydrophilic layer and as the adhesive material, thus obviating the need for applying another layer of adhesive material. Further variations will be apparent to the skilled artisan.

          “Waterproof” is determined by conducting waterproof testing as follows: Laminates are tested for waterproofness by using a modified Suter test apparatus, which is a low water entry pressure challenge. Water is forced against a sample area of about 4 ¼ inch diameter sealed by two rubber gaskets in a clamped arrangement. The sample is open to atmospheric conditions and is visible to the operator. The water pressure on the sample is increased to about 1 psi by a pump connected to a water reservoir, as indicated by an appropriate gauge and regulated by an in-line valve. The test sample is at an angle and the

water is recirculated to assure water contact and not air against the sample's lower surface. The upper surface of the sample is visually observed for a period of 3 minutes for the appearance of any water which would be forced through the sample. Liquid water seen on the surface is interpreted as a leak. A passing  
5 (waterproof) grade is given for no liquid water visible within 3 minutes. Passing this test is the definition of "waterproof" as used herein.

## **TEST METHODS**

### **COMPACTION**

10           Compaction volume of a pattern selectively compressed ePTFE laminate and non-selectively compressed samples are tested using ASTM Designation F 1853-98, Standard Test Method for Measuring Sleeping Bag Packing volume. The method quantifies and compares the packing volume of sleeping bags and like textile constructions under a standardized load. The standard cylinder used  
15 to test sleeping bags measures about 18 inches in diameter by about 32 inches high. However, for smaller test specimens, an about 5.5 inch diameter by about 31 inch high cylinder is used. The weight used to compress garment and laminate samples is about 25 pounds. The test sequence requires placing the test specimen within the circular round cylinder, and allowing it to settle, and when  
20 stable a plate that is slightly undersized, but closely matches the inside diameter of the measuring cylinder is placed on top of the garment, and the 25 pound weight load is lowered into place on top of the plate. The material is compressed by the weight. The height of the compacted material within the cylinder is measured by a scale affixed to the measuring cylinder.  
25 Measurements are taken of the compressed material at two location opposite to one another to the nearest 1/16 inch. The material is then removed, the laminate shaken out and allowed to relax. After several minutes it is replaced within the cylinder, and the test repeated. The four resulting height measurements are then averaged to yield the garment compaction height.

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### **WEIGHT**

Weight of samples are measured on a Mettler-Toledo Scale, Model 1060. The scale is recalibrated prior to weighing specimens. All weights are recorded in ounces to the nearest half ounce.

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### **HAND**

AATCC (American Association of Textile Chemists and Colorist) Evaluation Procedure 5 is used to measure the effect of selective compression on

the hand of ePTFE laminates by using a bending test. The equipment used is a Handle-O-Meter, Model 211-5-10 manufactured by Thwing/Albert Instrument Co. Philadelphia, PA. Ten test specimens of the desired material are cut to about 4 inch x about 4" squares. Five are cut in the fill direction. Five are cut  
5 in the warp direction. All specimens are then conditioned at  $70 \pm 2$  °F,  $65 \pm 2\%$  Relative Humidity (hereinafter "RH") for about 4 hours prior to testing. An about 1000 gram beam is used to push the test specimens through an about 1/4" slot. The resistance force, related to the bending stiffness of the fabric, is measured and displayed digitally. The peak force is recorded and used to  
10 compare samples. The samples are then averaged tested for hand using the Handle- O-Meter.

#### **MOISTURE VAPOR TRANSMISSION RATE TEST-- (MVTR)**

Samples are die-cut circles of 7.4 cm diameter. The samples are  
15 conditioned in a 23°C, 50%  $\pm 2\%$  RH test room for 4 hours prior to testing. Test cups are prepared by placing 15 ml of distilled water and 35 g of sodium chloride salt into a 4.5 ounce polypropylene cup, having an inside diameter of 6.5 cm at the mouth. An expanded PTFE membrane (ePTFE), available from W. L. Gore and Associates, Incorporated, Elkton, Maryland, is heat sealed to the lip  
20 of the cup to create a taut, leakproof microporous barrier holding the salt solution in the cup. A similar ePTFE membrane is mounted taut within a 5 inch embroidery hoop and floated upon the surface of a water bath in the test room. Both the water bath and the test room are temperature controlled at 23°C. The sample is laid upon the floating membrane, a salt cup is weighed, inverted  
25 and placed upon the sample. After one hour, the salt cup is removed, weighed, and the moisture vapor transmission rate is calculated from the weight pickup of the cup as follows:

$$\text{MVTR (grams/(m}^2\text{)(24 hours))} = \text{Weight (g) water pickup in cup} / [\text{Area (m}^2\text{) of cup mouth multiplied by the Time (days) of test}].$$
  
30

A combined moisture permeability is determined by one of two ways. The preferred way is to place the two adherends physically in contact, without adhesive, between the two ePTFE membranes of the test, as taught above. In this  
35 manner the adherends are positioned such that the measurement is a direct determination of the moisture vapor transmission rate of the adherends in series. There are certain situations where this configuration is not practical and as such the combined moisture permeability (used interchangeably with moisture vapor

transmission rate herein, MVTR) can be mathematically determined from the previously independently determined moisture transmission rate of the two adherends. This is accomplished by equating the sum of the reciprocals of the adherend MVTRs to the reciprocal of the combined MVTR and solving for the combined MVTR.

### **ABRASION**

The test used for abrasion is the Abrasion Resistance of Textile Fabrics Abrasion standard test method D. 4966-98 (Martindale Tester Method), by subjecting specimens to a rubbing motion against a piece of felt for 3,000 movements. The abraded samples are visually inspected for any change in aesthetics. Samples are preconditioned, then placed in a conditioned room at  $70^{\circ}\text{F} \pm 2^{\circ}\text{F}$  and  $65 \pm 2\%$  RH for at least four hours prior to testing. A piece of felt measuring about 5.5 inches square followed by a piece of the standard laminate of the same size is placed on the testing table. The machine mounting weight is placed on the table to flatten the felt/laminate samples. The felt/laminate is secured to the table with the mounting weight in place, then the weight is removed to inspect for tucks or ridges. The specimen is then placed face down into the specimen holder. The assembled holder is placed on the machine with a foam and wool abradent and the required weight is added to give pressure on each specimen. The amount of pressure is  $1.31 \pm 0.03$  psi. The counter is set to record the desired movements, and the machine started. After 3,000 movements, visual examination is done.

### **CURL**

Curl of desired fabric laminates is measured using Gore Test Method Fabla 00179. Test specimens are cut into about 5 inch by 5 inch squares. Three specimens are tested in the fill direction, and three in the warp direction. After cutting, specimens are taken into a conditioned lab, maintained at  $70 \pm 2^{\circ}\text{F}$ , and  $65 \pm 2\%$  RH for about four hours. The specimens are observed to detect any curl. If some curl is present, each specimen is placed with the curled side up on a flat surface away from drafts or air from fans. If no curl is obvious, each specimen with the film side up is placed onto a flat surface away from drafts or air from fans. If no curl is obvious on the fabric side, the sample is placed fabric side up on a flat surface away from drafts or air from fans. Specimens are allowed to lay undisturbed for about four hours.

After the about four hour equilibration period, each specimen is visually inspected and given a score of from 0 to 5. The direction of curl, if present, is

also recorded for both fill and warp specimens. A lower curl score indicates a sample that lies more flat. A curl score of 5 indicates a sample that will spontaneously roll up into a rod-like shape. A curl score of 0 indicates a sample that lies flat.

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### **GREY-SCALE**

The grey-scale of the micrographs of selectively compressed and of non-compressed control samples were analyzed via the following method.

Depending on the magnification required, the micrograph image was captured  
10 from either an optical microscope or a scanning electron microscope. The captured digital image was then converted to pixels. Image analysis software by EDAX was then used to create a frequency distribution of the grey-scale pixels.

### **3-DIMENSIONAL SURFACE MAP**

15 A 3-dimensional surface map of selectively compressed laminate samples was created using a Zygo Optical Surface Profilometer. A ten times objective lens was used and unless otherwise stated, a 100 micro bipolar distance was scanned. The resulting 3-dimensional surface map was then saved as a bit map for subsequent inclusion in other files.

20 The following non-limiting examples are provided to further exemplify aspects of the invention.

## **EXAMPLES**

### **Example 1**

25 A 2 layer laminate comprised of a brushed knit and an ePTFE film from W.L. Gore & Associates, Inc., of Elkton, MD, part number KAEX0010000D was selectively compressed using the following process. A 3 roll lab calendering system, from B.F Perkins of Rochester, N.Y. was used. The machine, having an about 22" face, was run at a speed of about 10 to about 15  
30 feet per minute, with floor mounted unwind and rewind, and a 2 zone fluid heating system. The rollers were heated to a temperature of about 240°F. A hardened metal leather patterned roll and a smooth, filled cotton mating roll were used. One sample of the laminate was run through with the ePTFE surface facing the patterned roll. A second sample of the laminate was run through with  
35 the fabric surface facing the patterned roll. In both cases, the image of the pattern was transferred onto the ePTFE film side of the 2 layer laminate. Both appearance and texture had been changed. The ePTFE surface had the appearance of the leather pattern of the roll.

Figure 4 is an optical micrograph of the ePTFE side of the laminate, after selective compression. The selectively compressed regions of the ePTFE appear to be darker in color, but in fact are more translucent and show the color of the textile on the opposite side of the ePTFE. The non-compressed regions are clearly shown to be the known “white” color of non-compressed ePTFE.

### **Example 2**

A 2 layer laminate comprised of a brushed knit and an ePTFE film from W.L. Gore & Associates, Inc., of Elkton, MD, part number KAEX00100D was selectively compressed using the following process. A 2 roll production calendering system at Lee Fashion Fabrics, Inc. of Johnstown, N.Y. was used. The machine, having an about 56 inch face, was run at a speed of about 30 feet per minute. The rolls were heated to about 350°F. A hardened metal paisley patterned roll and a smooth, hard rubber roll were used. One sample of the laminate was run with the ePTFE surface facing the patterned roll. A second sample of the laminate was run with the fabric surface facing the patterned roll. In both cases, the image of the pattern was transferred onto the ePTFE film side of the laminate. Figure 5 is an optical micrograph of the ePTFE film side of the laminate. Both appearance and texture had been changed. The ePTFE surface had the appearance of the paisley pattern of the roll. As in Example 1, the selectively compressed regions of the ePTFE film appear to be darker, but in fact are more translucent and show the color of the textile material on the opposite side of the ePTFE. The non-compressed regions are clearly seen to be the known “white” color of ePTFE.

### **Example 3**

A 2 layer laminate comprised of a brushed knit and an ePTFE film from W.L. Gore & Associates, Inc., of Elkton, MD, part number KKRX620000 was selectively compressed using the following process. A 2 roll production calendering system at Lee Fashion Fabrics, Inc. of Johnstown, N.Y. was used. The machine, having an about 56 inch face, was run at a speed of about 30 feet per minute. The rolls were heated to about 350°F. A hardened metal leather patterned roll and a smooth, hard rubber roll were used. One sample of the laminate was fed through with the ePTFE surface facing the patterned roll. A second sample of the laminate was fed through with the fabric surface facing the patterned roll. In both cases, the image of the pattern was transferred onto the ePTFE film side of the laminate, as depicted in the optical micrograph shown in Figure 6. Both appearance and texture had been changed. The ePTFE surface had the appearance of the leather pattern of the roll.



The various depths of the image on the patterned roll produced a surface image and pattern of varying degrees of translucency. Figure 7 is a grey-scale spectra that depicts the range of shades that were effectively imparted to the formerly white ePTFE film-side of the laminate.

5    **Example 4**

A 2 layer laminate comprised of a knit and an ePTFE film from W.L. Gore & Associates, Inc., of Elkton, MD, part number KKRX620000 was selectively compressed using the process and machine described in Example 2. The roller temperature was about 350°F. The machine was run at a speed of  
10    about 30 feet per minute. A hardened metal, stripe-patterned roll and a smooth, hard rubber roll were used for this example. A sample of the laminate was fed with the ePTFE surface facing the patterned roll. A second sample of the laminate was fed with the fabric surface facing the patterned roll. The image of the stripe pattern was transferred onto the laminate. When the white ePTFE film  
15    side was facing the patterned roll, the image transferred to the film appeared crisp and sharp. When the knit faced the patterned roll, the image transferred to the ePTFE film and while still very clear, was slightly more diffuse and subtle. Figure 8 is an optical micrograph that shows the surface image created by the stripe pattern selective compression. Figure 9 shows the corresponding grey-  
20    scale frequency distribution highlighting the fact that the translucency varies with the degree of selective compression. Figure 10 shows an SEM cross-section micrograph of the transition between the compressed and non-compressed areas. Figure 11 shows a 3-dimensional surface map depicting the various depths of selective compression that resulted. Figure 12 shows a Zygo  
25    profilometer scan of the surface of a single stripe of this stripe patterned selectively compressed laminate sample.

**Example 5**

A 2 layer laminate comprised of a knit and an ePTFE film from W.L. Gore & Associates, Inc., of Elkton, MD, part number KKRX620000 was  
30    selectively compressed using the process and machine described in Example 2. The roller temperature was about 350°F. The machine was run at a speed of about 30 feet per minute. A hardened metal, snake skin-patterned roll and a smooth, hard rubber roll were used. One sample of the laminate was fed with the ePTFE surface facing the patterned roll. A second sample of the laminate  
35    was fed with the fabric surface facing the patterned roll. The image of the linen pattern was transferred onto the laminate. When the white ePTFE film side was facing the patterned roll, the image transferred to the film appeared crisp and

sharp. When the knit faced the patterned roll, the image transferred to the ePTFE film and while still very clear, was slightly more diffuse and subtle.

#### **Example 6**

5 A 2 layer laminate comprised of a knit and an ePTFE film which was coated with a breathable polyurethane layer from W.L. Gore and Associates, part number WNAX467000 was selectively compressed using the process described in Example 1, using the same roller temperature and line speed. A hardened metal, linen-patterned roll and a smooth, hard rubber roll were used. One sample of the laminate was fed with the ePTFE surface facing the patterned  
10 roll. A second sample of the laminate was fed with the fabric surface facing the patterned roll. The image of the linen pattern was transferred onto the laminate. When the white ePTFE film side was facing the patterned roll, the image transferred to the film appeared crisp and sharp. When the knit faced the patterned roll, the image transferred to the ePTFE film and while still very clear,  
15 was slightly more diffuse and subtle.

#### **Example 7**

A 2 layer laminate comprised of a brushed knit and an ePTFE film from W.L. Gore & Associates, Inc., of Elkton, MD, part number KAEX00100D was selectively compressed using the following process. A 3 roll lab calendering  
20 system, from B.F Perkins of Rochester, N.Y. was used. The machine, having a 22" face, was run at a speed of about 10 to about 15 feet per minute, with floor mounted unwind and rewind, and a 2 zone fluid heating system. A smooth, hardened metal roll and a smooth, filled cotton counter roll were used. One sample of the laminate was fed with the ePTFE surface facing the metal roll. A  
25 second sample of the laminate was fed with the fabric surface facing the metal roll. In both cases, the yarns of the knit material caused the ePTFE film to be selectively compressed so that the knit pattern was effectively transferred onto the ePTFE film of the laminate.

#### **Example 8**

30 A 3 layer laminate comprised of a knit material on each side of a two layer ePTFE film construction, between which was sandwiched a breathable polyurethane layer, was selectively compressed. One side of the 3 layer laminate had a lighter weight knit than the opposite side (W.L. Gore and Associates, part number KPBX602607). The 3 layer laminate was selectively compressed using  
35 the process described in Example 2, using the same roller temperature and same line speed. A hardened metal, snake skin-patterned roll and a smooth, hard rubber roll were used. The laminate was fed through the rollers with the

lighter-weight knit surface facing the patterned roll. The image of the snake-skin pattern was transferred onto the 3 layer laminate.

**Example 9: Compaction tests**

Three selectively compressed laminate samples and their respective  
5 controls were tested for compaction using the test method described above.  
Sample 1 was a three layer ePTFE containing laminate (Gore Part  
#WNAX002604A). The control for Sample 1 was a non-compressed laminate  
of this material. Sample 1 was produced by taking a sample of this laminate and  
selectively compressing the laminate as described in Example 2, using a snake  
10 skin patterned roll. Sample 2 was a two layer ePTFE containing laminate (Gore  
Part #WKPX003000). The control for Sample 2 was a non-compressed  
laminate of this material. Sample 2 was produced by taking a sample of this  
laminate and selectively compressing the laminate as described in Example 2.  
Sample 3 was a three layer ePTFE containing laminate (Gore Part #  
15 KPBX602607). The control for Sample 3 was a non-compressed laminate of  
this material. Sample 3 was produced by taking a sample of this laminate and  
selectively compressing the laminate as described in Example 2.

Each laminate was then cut to a size of about 72 inches by about 52  
inches to produce samples for compaction testing. Each laminate sample was  
20 lowered into the test cylinder, allowed to settle for about 1 minute. When stable,  
weights measuring about 25 pounds were lowered on top of the plate. The test  
specimen was compressed by the weight. The height of the compacted test  
specimen within the cylinder was measured by a scale affixed to the measuring  
cylinder. Measurements of the height of the compressed test were taken at two  
25 locations opposite to one another, with height measurements being to the nearest  
1/16 inch. As discussed above, the test was repeated for each sample and the  
measurements averaged to yield the reported Compacted Height.

Table 1

30

Sample	Selective Compression Process	Control Sample Compacted Height (inches)	Selectively Compressed Sample Compacted Height (inches)
Sample 1	Compressed as described in Example 2	2.75	2.50
Sample 2	Compressed as described in Example 2	3.00	2.63
Sample 3	Compressed as described in Example 2	3.13	2.75

**Example 10 – Hand**

Following the procedure described in Example 2 above, a 3 layer ePTFE laminate (Gore Part #WNAX002604A) was selectively compressed. The hand of this test specimen was compared to the control laminate (non-compressed Gore Part # WNAX002604A). A lower hand score indicates a softer sample. The control laminate registered a hand of 194 and the selectively compressed laminate registered a hand of 134.

**Example 11 – Curl**

Following the procedure described in Example 2 above, a lightweight 2 layer ePTFE laminate (Gore Part # ASND 152000P3) was selectively compressed. The curl of this test specimen was compared to the control (non-compressed Gore Part #ASND 152000P3) laminate. The control laminate was judged to have a curl value of 0 and the selectively compressed laminate was judged to have a curl value of 4.

**Example 12 – Breathability and waterproofness results**

Six laminate materials were selectively compressed as described in Example 2. The selectively compressed laminates and control laminates (the same laminate, but not selectively compressed) were tested for Water Vapor Transmission Rate (i.e. breathability) using the test method described above. Each laminate sample was tested before and after selective densification. Moreover, each selectively compressed laminate was tested for waterproofness. The results are shown in Table 2.

Table 2

Sample	Pattern used to impart selective compression	Control MVTR (g/(m <sup>2</sup> )(24 hours))	Selectively Compressed MVTR (g/(m <sup>2</sup> )(24 hours))	Waterproof Testing Results
2 Layer ePTFE laminate Gore part # KKRX620000	Leather	13,859	12,029	Pass
2 Layer ePTFE laminate Gore part # KKRX620000	Stripe	13,859	12,533	Pass
2 Layer ePTFE laminate. Gore part # WNOX117000	Snake-skin	14,604	12,278	Pass
3 Layer ePTFE laminate. Gore part # KPBX602607	Snake-skin	8,303	7,840	Pass
3 Layer ePTFE laminate. Gore part # WANX002604A	Snake-skin	7,406	6,058	Pass
WINDSTOPPER® brushed knit laminate. Gore part # KAEX00100D	Paisley	19,241	16,178	Pass

### Example 13 – Abrasion results

5           A first sample of a two layer ePTFE containing laminate (Gore Part #KAEX00100D) was selectively compressed as described in Example 2, using a paisley patterned roll. A second sample of the same laminate was not selectively compressed. Each sample was then subjected to the Abrasion test described above (i.e. Abrasion Resistance of Textile Fabrics Abrasion standard  
10 test method D. 4966-98 (Martindale Tester Method)), with the ePTFE side of each sample being subjected to abrasion.

Optical micrographs of a section of each sample were then taken. Figure 13 is an optical micrograph of a section of the sample that was not selectively compressed. The Figure clearly shows the stark contrast between a portion of the sample that was subjected to the abrasion testing (i.e. the darker portion of the sample) and a portion of the sample that was not subjected to abrasion  
15 testing (i.e. the lighter portion of the sample). Figure 14 is an optical micrograph of a section of the sample that was selectively compressed. As can

be seen, the paisley pattern of the sample is still present. Although there is still a contrast between the portion of the sample that was subjected to the abrasion testing and the portion of the sample that was not subjected to the abrasion testing, the contrast is not as stark as in the non-selectively compressed sample.

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